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AN INVESTIGATION OF THE STRUCTURE AND PHASE COMPOSITION
OF A SILICON DIFFUSION COATING FOR NIOBIUM

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AN INVESTIGATION OF THE STRUCTURE AND PHASE COMPOSITION OF A SILICON
DIFFUSION COATING FOR NIOBIUM

by

P.M.Arshanyy, R.M.Volkova, D.A.Prokoshkin

Niobium falls into the category of refractory metals (tungsten, molybdenum, tantalum) but differs advantageously from them by a combination of special properties: high strength and ductility at room and elevated temperatures, relative low specific gravity, nonvolatility of oxides at high temperatures and good susceptibility thereof to manufacturing processes. Niobium is also of interest for nucleonic engineering, inasmuch as, at high temperatures, its thermoneutron capture ~~may~~ cross section is lower than that of any other metal of analogous strength. Moreover, niobium is distinguished by good corrosion strength in virtually all chemicals other than hydrofluoric acid. It is true however, that the stability of niobium in acids diminishes with rise in temperature.

Gases, particularly oxygen and nitrogen, have a powerful effect upon niobium. With increase in temperature, the solubility of gases in niobium rises, and this is reflected in a reduction of the melting point of pure niobium. Oxygen and nitrogen substantially increase the strength of niobium and the metal becomes hard and brittle at a particular content thereof.

The shortcomings of niobium include its low resistance to corrosion in the air at high temperatures. A rise in the corrosion-resistant properties of niobium at elevated temperatures is achievable by thermodiffusion coating with various elements. We have performed an investigation of the structure and phase composition of technical niobium saturated ~~with~~ with solid-phase silicon for 30 min to 15 hrs at temperatures of 1100 - 1300°.

Analysis of the coating shows that it consists of a series of layers separated by boundaries. The broad bright outer layer which, according to x-ray spectral analysis contains about 37% Si, is a silicide NbSi_2 with ^{hexagonal} ~~orthorhombic~~ lattice and the following parameters $a = 4.788 \text{ KX}$, $c = 6.58 \text{ KX}$.

The microhardness of the layer is high (1050 kg/mm^2).

The second layer is thin, also bright and weakly etched, and consists of about 14% of the total thickness of the layer and contains about 15% silicon. Its microhardness is somewhat lower (700 kg/mm^2). The layer is a combination of three modifications of ~~NbSi~~ Nb_5Si_3 : one with an α -tetragonal lattice: $a = 6.58 \text{ KX}$, $c = 11.87 \text{ KX}$, another with a γ -hexagonal lattice: $a = 7.51 \text{ KX}$, $c = 5.29 \text{ KX}$; a third with a β -tetragonal lattice: $a = 10.02 \text{ KX}$, $c = 5.10 \text{ KX}$.

The third layer, which is separated from the others by a rather ^{clear} ~~sharp~~ boundary contains, according to data of x-ray spectral analysis, about 7% silicon and represents the compound Nb_4Si . We have not determined its crystalline structure.

Figure 3 presents radiographs taken from standard specimens of silicon-saturated niobium. The change in the duration of saturation of niobium with silicon does not result in changing the microstructure or the phase composition of the coating. The relationship between the thicknesses of the various layers of the coating is retained despite the changes in the duration of saturation.

The relationship between the weight ^{increment} ~~gain~~ Δp to time τ is presented in Fig. 4, where a curve replotted in log-log scale, yields a linear course and the value of angle $\alpha = 26^\circ 30'$, indicating that there is a quadratic parabolic relationship.

~~THE~~ Below we present data characteristic of the change in the weight gain Δp of the specimens relative to process ~~EXT~~ temperature with constant holding time of 6 hrs:

Temperature, $^\circ\text{C}$

Δp , mg/cm^2

This change in the relationship of weight gain to temperature is exponential.

It follows from the foregoing that the weight of the specimens increases that much more, the higher \nearrow the temperature and the greater the duration of the saturation process. Thus, in the process of diffusive saturation of niobium with silicon, a coating of variable composition is established. Tests have shown that it is chemically stable but brittle and that it may be destroyed by stress at high temperatures.

We have computed the diffusion constants of silicon in niobium for the NbSi_2 and

the Nb_5Si_3 phases, which yielded clearly)

marked interface boundaries and which were present at all the temperatures studied.

The condition of application of the parabolic law - constancy of composition at the

boundaries - was fulfilled best under these

conditions. Φ The temperature relationship of

the coefficient of diffusion is subordinate

to an exponential ~~XX~~ law and is described by)

the
formula:

Fig.2 - Distribution of Concentration
of Silicon, Relative to Depth of
Diffusion Layer

a) Distance from surface l , microns

or

(1)

where y is the thickness of the layer in

microns. After we have found the logarithm of eq.(1), a linear relationship is revealed between the experimental data when they are represented in coordinates consisting of $\log y$ and $\frac{1}{T}$. The tangent of the slope to $\log y$ to the $\frac{1}{T}$ axis determines the heat of the energy of activation.

As we know, various methods are employed to compute the major parameters of

Fig.3 - Radiographs of Silicide Compounds of Niobium:

- a - Standard of comparison from the surface of a specimen NbSi_2 ;
b - NbSi_2 ; c - At 50 microns depth $\text{NbSi}_2 + \text{Nb}_5\text{Si}_3$; d - At 110 microns
depth; e - Standard of comparison Nb_5Si_3 ; f - Nb base

203 diffusion, including graphic methods, algebraic methods and the method of least squares, regarded which is ~~known~~ as the most perfect. In the present paper, the computation of the ~~known~~ pre-exponential factor D_0 and the ~~known~~ activation energy Q was performed by all three methods. The results show that they are similar to each other in absolute data. The data derived are set forth in the Table.

205 Parameters of Diffusion of Silicon in Niobium

mg/cm^2

τ, hrs

Change in Weight
Fig.4 - ~~known~~ Gain of Specimens Relative to Time
Allowed for Saturation at Temperature 1200°
a - In coordinates of Δp versus τ ; b - In log-log coordinates

Summary

1. The diffusion of silicon into niobium has been studied in relationship to temperature and process time. The parameters of diffusion of silicon into niobium D_t , D_0 and Q have been computed.

2. At saturation temperature up to 1300°, phases of the following composition are developed on the surface of the niobium: $NbSi_2$, Nb_5Si_3 , and Nb_4Si .

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